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Client-Matter Number
SHIG C11119

February 18, 2004

TELECOMMUNICATION INFORMATION COVER SHEET

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FROM: Norman P. Soloway

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MESSAGE:

Re: OKADA et al.

U.S. Patent Application Serial No. 09/913,625

Filing Date: August 16, 2001

For: HYDROGEN STORAGE METAL ALLOY AND ...

Our Ref: SHIG C11119

Transmitted herewith:

1. Transmittal Letter (2 pgs); and
2. Copy of Declaration under 37 CFR 1.132 w/ accompanying materials (13 pgs).

2/18/04 sb

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re Appln. of: OKADA et al.

Serial No.: 09/913,625

Filed: AUGUST 16, 2001

For: HYDROGEN STORAGE METAL ALLOY AND METHOD...

Group: 1742

Examiner: IP, SIKYIN

DOCKET: SHIG C11119

Commissioner for Patents
P.O. Box 1450
Alexandria, VA 22313-1450

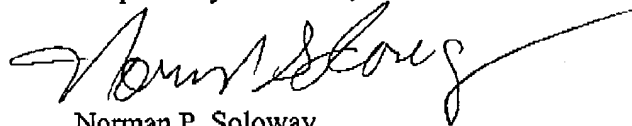
TRANSMITTAL LETTER

Dear Sir:

In connection with the above-entitled matter, please find a copy of the previously submitted Declaration under 37 CFR 1.132 together with the several attachments mentioned therein.

In the event there are any fee deficiencies or additional fees are payable, please charge them (or credit any overpayment) to our Deposit Account No. 08-1391.

Respectfully submitted,



Norman P. Soloway
Attorney for Applicants
Reg. No. 24,315

HAYES SOLOWAY P.C.

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Serial No. 09/913,625
Docket No. SHIG C11119
TRANSMITTAL LETTER

CERTIFICATE OF TRANSMISSION VIA FACSIMILE

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Sikyin Ip of the United States Patent and Trademark Office at facsimile number
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By 

NPS:sb

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FEB 18 2004

OFFICIAL

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re Appln. of: OKADA et al.
Serial No.: 09/913,625
Filed: AUGUST 16, 2001
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Examiner: IP, SIKYIN DOCKET: SHIG C11119
Commissioner for Patents
P.O. Box 1450
Alexandria, VA 22313-1450

DECLARATION UNDER 37 CFR 1.132

The undersigned, being the named inventors of the above identified Application, declare and state as follows:

- (1) This Declaration is being filed in further support of our above identified U.S. Application. Our CV's are attached.
- (2) We understand our Application has been rejected as being obvious from Japanese Application No. JP 10-121180 (JP '180), taken alone or in combination with JP 11-106859. In particular, we understand the Examiner has cited JP '180 as teaching the advantages of quenching in iced water. We respectfully disagree. While JP '180 may mention quenching in iced water, nowhere within the four corners of JP '180 do the authors teach the advantages of quenching in iced water. Indeed, the working Examples in JP '180 do not use iced water.
- (3) Quenching in iced water is critical to the practice of our invention. To demonstrate the criticality of quenching in iced water, we conducted a series of experiments

Serial No. 09/913,625
Docket No. SHIG C11119
Declaration under 37 CFR 1.132

comparing the results of oil-quenching and quenching in iced water after heat treatment. The results are reported in Graphs 1 and 2 attached hereto, wherein:

Graph 1 shows X-ray diffraction patterns of Ti-57.5Cr-2.5 Mo alloys (oil-quenched or iced water-quenched after heat treatment). From Graph 1 it is shown that the iced water-quenched alloy has only clear BCC peaks while an oil-quenched alloy is contaminated with C15 & C36 Laves.

Graph 2 shows PTC curves of Ti-57.5Cr-2.5Mo alloys oil-quenched or iced water-quenched after heat treatment). From Graph 2, it is shown that the iced water-quenched alloy provides higher protium concentration (mass%) values, about 3.4 to 3.5 mass%, than an oil-quenched alloy.

(4) We have discovered that "rapidly cooled preferably at a cooling rate of more than 100K/sec." (page 19, lines 31 to 32 of the Applicants' sworn specification) is critical. The hydrogen storage alloys of the present invention have unexpectedly higher hydrogen storage capacities (see FIGS. 9 & 10). In FIGS. 9 & 10 of the subject Application, each storage capacity of the instant alloys is over 2.6 mass% within a range of both $0 < \text{Mo at\%} < 5$ and $0 < \text{W at\%} < 5$.

(5) A difference of 0.1 to 0.2 mass% per unit alloy mass is a remarkable value in this field in connection with Ti-Cr-system alloys having a quite low level of Mo and/or W. The increase of even 0.1 to 0.2 mass % in the hydrogen storage capacity is an unexpected, superior effect over the prior art alloys. This difference is derived from the difference between prior art

Serial No. 09/913,625
Docket No. SHIG C11119
Declaration under 37 CFR 1.132

alloy treatment processes and the instant unique ones, and reflects the greatly different lattice structures (i.e., highly BCC-rich structures).

(6) There are other distinctions. JP '180 fails to teach or suggest a "homogenizing" treatment as required by step (2) of claim 22 of the present invention. The prior art technical concept with faults leads to "The preferred range of Mo and/or W is 5% or more but 20% or less" ([0012] on page 3, column 4, lines 8 to 10 of JP '180).

(7) Also critical to our invention is the discovery that the highly elevated temperature is effective in forming BCC-rich alloys and the heat treatment, i.e. of 1400°C or higher, has to carry out within a very short period of time. In the working Examples of Applicants' sworn specification, all the alloys are produced at 1400°C or higher. -- As demonstrated in the attached Graphs 3 and 4, a short time heat treatment is preferable:

Graph 3 shows hydrogen absorption and desorption characteristics of Ti-57.5 Cr-2.5 Mo alloy where the heat-treatment time varies at 1400°C; and

Graph 4 shows PTC curve profiles of Ti-57.5 Cr-2.5 Mo alloy where the heat-treatment time varies at 1400°C.

From Graphs 3 and 4 it is demonstrated that the prolonged heat-treatment deteriorates the hydrogen absorption and desorption characteristics of hydrogen storage alloy products. See also Cr-Mo-Ti state diagrams, Graph 5.

(8) We have found that it is extremely important to:

(1) repeatedly melt and solidify the starting mixture to form a heated homogeneous alloy,

Serial No. 09/912,623
Docket No. SHIG C11119
Declaration under 37 CFR 1.132

(2) maintain the resultant heated homogeneous alloy at a temperature within a range just below the melting point of the alloy (i.e., 1400°C or higher) for a predetermined time (rather a shorter period) of from 1 to 100 minutes, and

(3) rapidly cooling the alloy from the above step (b) in iced water, and the combination of the above steps results in the formation of unexpected BCC-rich (or high hydrogen storage capacity) alloys even at a quite low level of Mo and W.

(9) Thus, it is shown that the combination of repeatedly melting and solidifying the starting mixture to form a heated homogeneous alloy, maintaining the heated homogeneous alloy at a temperature above 1400°C for from 1 to 100 minutes, and rapidly cooling the alloy in iced water results in hydrogen storage alloys having unique properties not achievable by the prior art.

(10) We hereby declare that all statements made hereof of our own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

Date: Jan. 8, 2004

By: Masuo Okada
Masuo OKADA

Date: Jan. 8, 2004

By: Takahiro Kuriwa
Takahiro KURIWA

Resume

Name: Masuo Okada
Date & July 5, 1948,
Place of Birth: Tochigi-Prefecture, Japan
Marital Status: Married, Two children
Nationality: Japanese

Degrees:	Date	Institution
Bachelor of Science in Materials Science	March, 1971	Tohoku University
Master of Science in Materials Science	Sept., 1974	Univ. of California, Berkeley, U.S.A.
Master of Science in Materials Science Sendai, Japan	Sept., 1975	Tohoku University
Doctor of Philosophy in Materials Science	June, 1978	Univ. of California, Berkeley, U.S.A.

Thesis:

Ph.D Microstructure and Magnetic Properties of Spinodal
Fe-Cr-Co alloys
Supervisor: Professor Gareth Thomas

Positions:

Research Associate: April 1979- November 1981
At the Department of Materials Science, Tohoku University, Sendai
Japan 980
Associate Professor: November 1981- April 1993
ibid
Professor: May 1993-Present

Research Activities

Prof.Okada's research activities address new developments of a wide range of functional materials, such as hydrogen-absorbing materials, electroceramics, and magnetic materials.

Recently, he served as a leader of the Japanese national project "New Protium Function in Materials" to explore new, useful functions of the hydrogen atom in materials, with the budget of about 6.5million dollars, supported by the Japanese Ministry of Education, from 1998-2001. The project was finished with great success. The evaluation committee of the national project gave the highest evaluation of A to this project. One of the distinguished results was that Professor Okada successfully developed the Ti-Cr-V or Ti-Cr-Mo hydrogen absorbing alloys with 3mass% of hydrogen operated below 100°C, which is still world record up to now. He gave the invited talks on these Ti-Cr-V or Ti-Cr-Mo alloys at the International Symposium of Metal-Hydrogen, which is most prestigious international conference for hydrogen related materials, at Noosa in Australia on October of 2000, and at Annecy in France on September of 2002. . Because of his outstanding achievements in the field of developments of new hydrogen absorbing materials, he is regarded as one of the world's outstanding researchers in hydrogen societies.

Resume

Name: Takahiro Kuitwa
Maritus Status: single
Nationality: Japanese

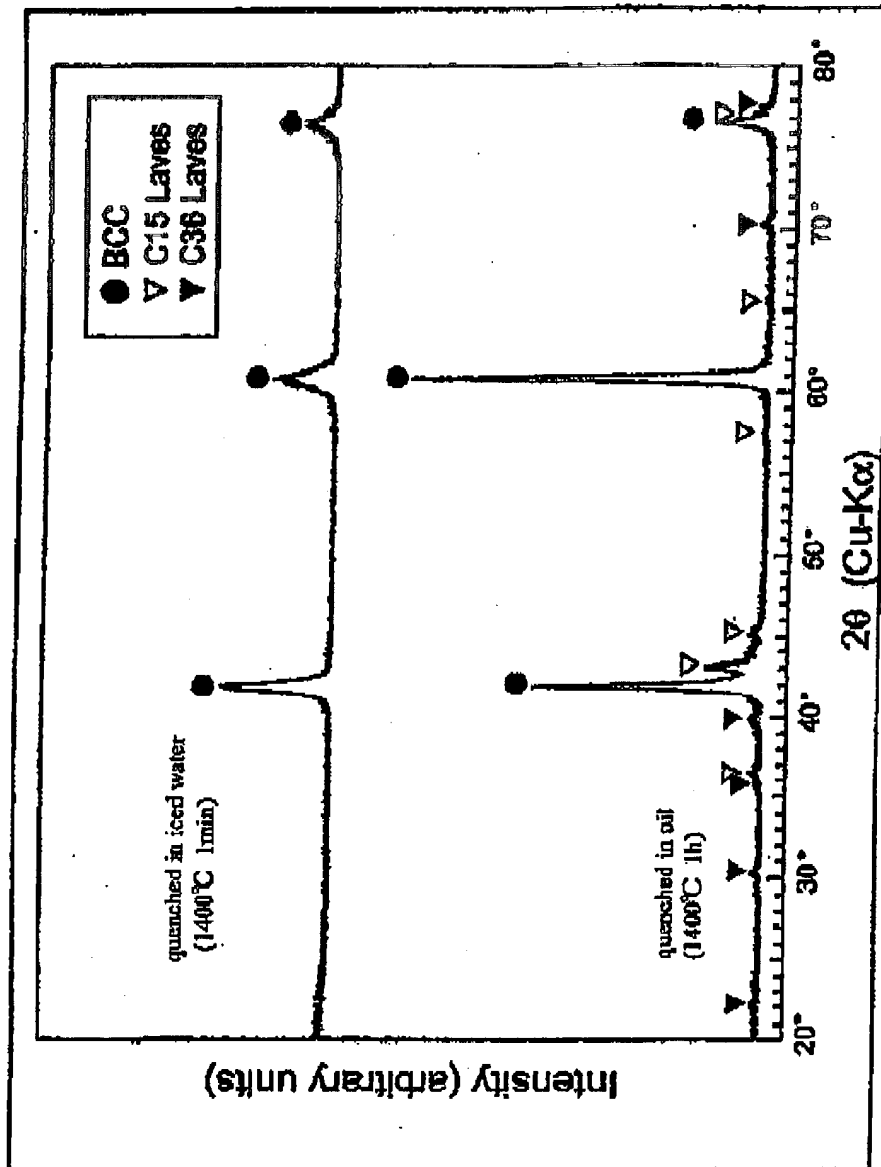
Degrees:	Date	Institution
Bachelor of Science in Materials Science	March, 1994	Tohoku University
Master of Science in Materials Science	March, 1997	Tohoku University
Doctor of Philosophy in Materials Science	March, 2000	Tohoku University Sendai, Japan

Thesis:

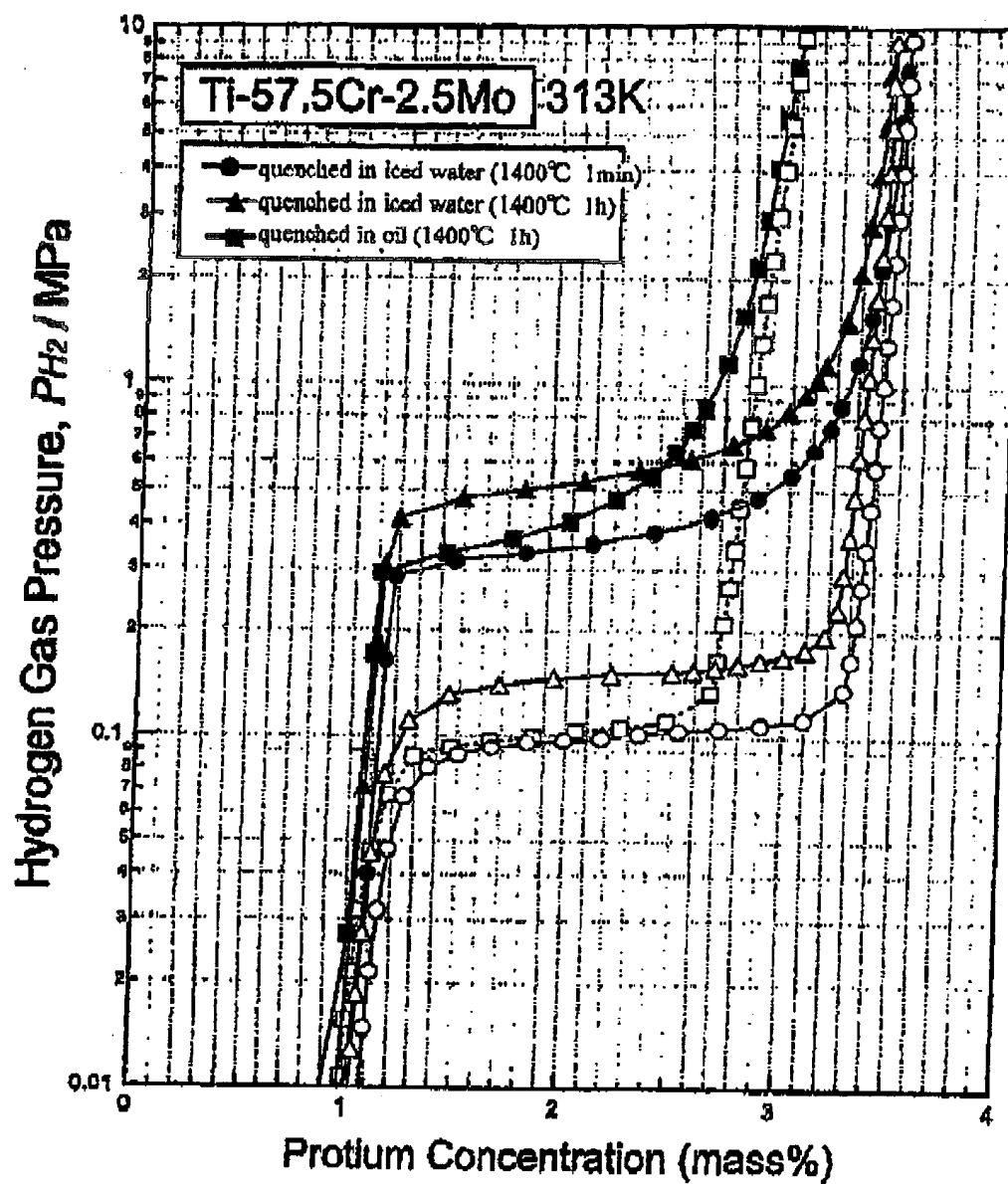
Ph.D Developments of V-based Hydrogen Absorbing Alloys with High
Content of Hydrogen
Supervisor: Professor Masuo Okada

Positions:

Research Engineer
Engineering Research Department
Honda R&D Co.Ltd. Tochigi R&D Center
Tochigi, Japan

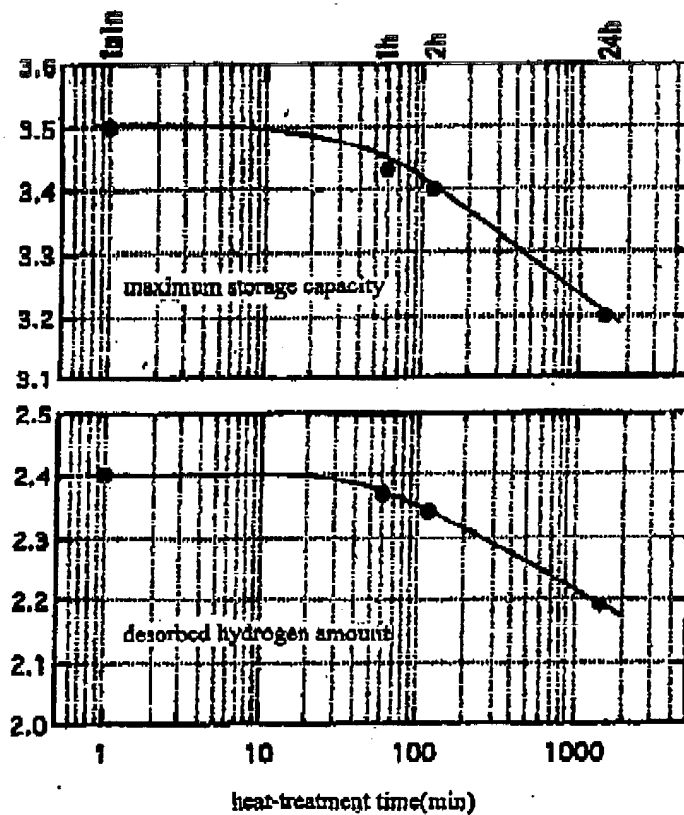


Graph.1
X-ray diffraction patterns of Ti-57.5Cr-2.5Mo alloys
(oil-quenched or ice water-quenched after heat treatment)

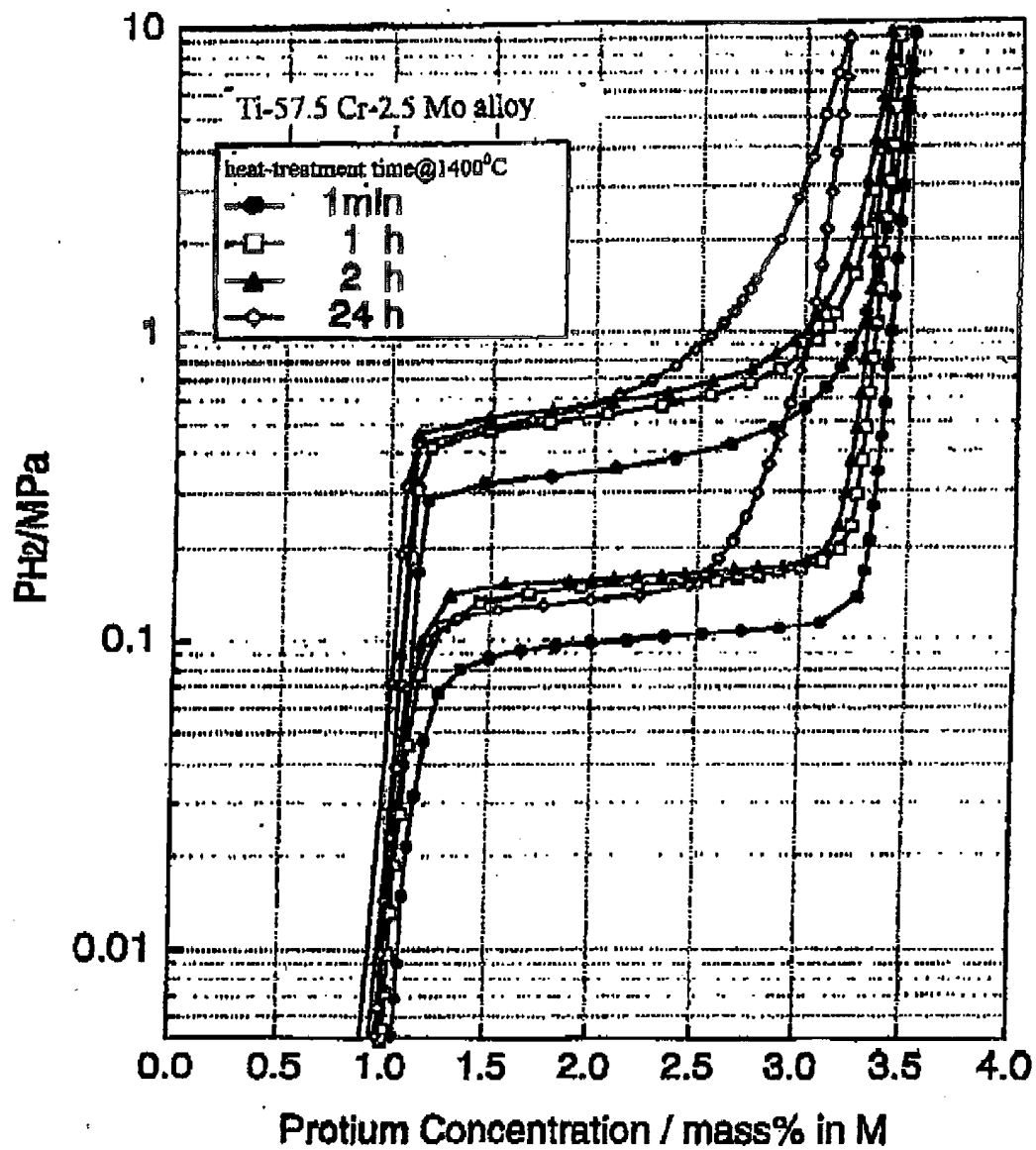


Graph.2
PTC curves of Ti-57.5Cr-2.5Mo alloys
(oil-quenched or iced water-quenched after heat treatment)

Ti-57.5 Cr-2.5 Mo alloy



Graph.3
hydrogen absorption and desorption characteristics of
Ti-57.5 Cr-2.5 Mo alloy where the heat-treatment time varies at 1400°C

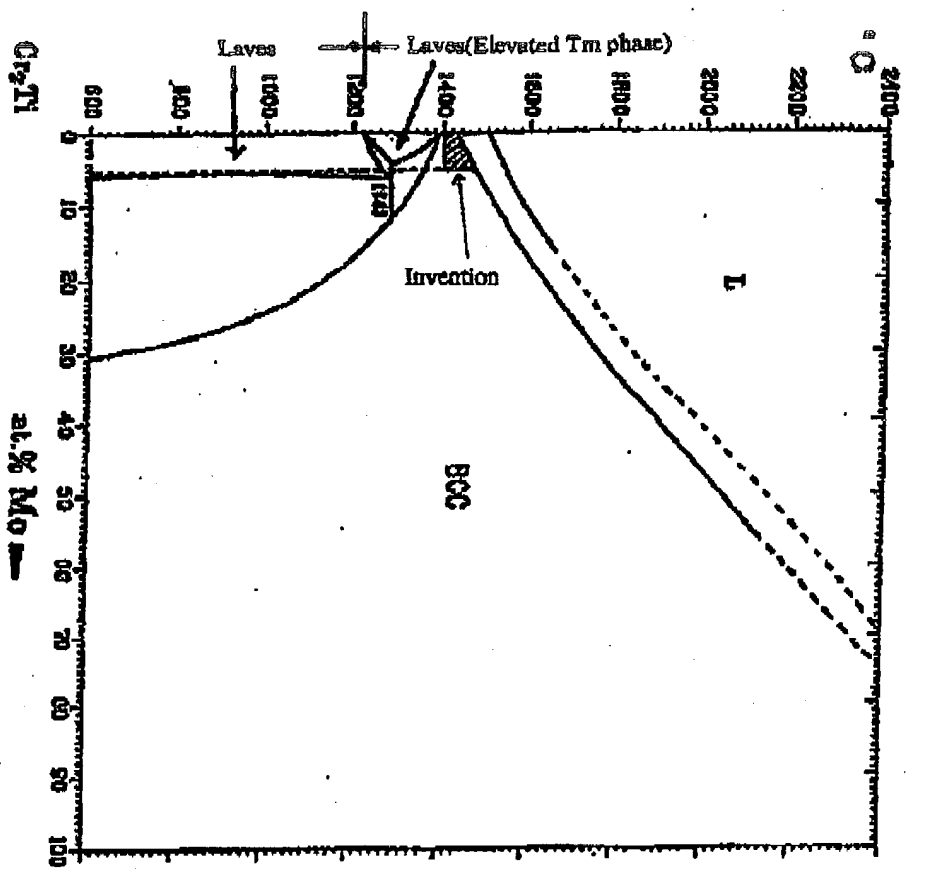


Graph.4

PTC curve profiles of Ti-57.5 Cr-2.5 Mo alloy
where the heat-treatment time varies at 1400°C

L1/OJ
at.% CoNi

Cr-Mo-Ti



Mo [Graph. 5.]

第4期

Hydrogen Fuel Cell Letter June 1999

card, docking and refueling, undocking after issuing a receipt - is extremely quick: docking and undocking take only a few seconds, according to the project description, and filling the tank with 120 liters of LH₂ takes about the same time as it takes to fill up with gasoline - about one and a half minutes in most recent tests. The car's range is more than 300 km (185 miles).

The robot arm with its coupling, coupling activation elements, docking sensor and opening and closing mechanism moves in four axes and connects to the car's fuel port anywhere within an envelope of 1 x 0.8 x 0.2 meters.

The station's control and measurements systems were designed by Siemens Automation and Drives Division, based on the company's Simatic S7 design. The Hydrogen monitoring system was contributed by Grinnel Laboratory Technology. Safety of the entire system was certified by the southern German section of TÜV, the national technical safety organization.

The project is scheduled to operate for at least two years until 2001 when the results will be evaluated, but it may continue for another two years beyond that. Contact: B&W (media), Thomas Steffer, phone 049/89/382-24697, fax 382-20626, e-mail thomas.steffer@bwm.de; airport (media), phone 049/89/973-41100, e-mail sp@munich-airport.de. □

BRIEFLY NOTED:

* The Bonneville Power Administration (BPA), Portland, OR has contracted to buy 110 fuel cells from Northwest Power Systems, Bend, OR for testing. BPA will take delivery of the first "alpha" 3 kW PEM systems for home use this fall, with the other "beta" units, which may incorporate needed changes, to be delivered later. The systems will be placed in the homes of interested customers in cooperation with local utilities. The beta units are expected to cost about \$30,000 each which may drop to less than \$10,000 once produced in commercial volumes by 2002, the release quoted NPS president Alan Guggenheim as saying. Contact: Perry Gruber, BPA (media), phone 503/730-5103, Alan Guggenheim, NPS, phone 541/583-3590.

* Avista Labs, Spokane, WA have selected Logan Industries, Inc., Spokane, as contract manufacturing partner for its introductory PEM fuel cell generators (H&FCL Dec. 98) slated for field testing this spring. Logan will manufacture, assemble and test a minimum of 200 systems, with first deliveries scheduled for the end of May. Avista Labs is a subsidiary of Avista Corp., a diversified energy company. Contact: Avista web site www.avistacorp.com.

* DoE's Ames Laboratory, Ames, IA has received \$750,000 over three years to start building a prototype magnetic refrigeration cooling unit in cooperation with Milwaukee-based Astronautics Corp. of America, the laboratory announced. Both groups have signed a Cooperative Research and Development Agreement to develop a rotary prototype. Project coordinator is Karl

Gschneidner, Jr., of Ames Laboratory. Ames and Astronautics built a proof-of-principle model in 1995, and two years ago the laboratory announced the development of new magnetocaloric materials (H&FCL July 97). Contact: Karl Gschneidner, Ames, 515/294-7931, Astronautics, Carl Zimm, 608/221-9001.

* SatCon Technology Corp., Cambridge, MA has received an additional order for electric motors and controllers for use in fuel cell vehicles from Opel, General Motors' German subsidiary. The company's advanced integrated power module controller, a variant of the PEBB controller supplied to Ping Power for residential fuel cell systems, is expected to sell for less than \$500 per unit, according to the release. Contact: Michael Turmelle, SatCon, 617/661-0548.

* Photofabrication Engineering, Inc., Milford, MA, says it can now photo-etch pure titanium for use in cathodes and anodes in fuel cells, oxygen generators and hydrogen generators. The company says its proprietary process can chemically etch titanium from extremely thin (0.0005 in.) to extremely thick (0.090 in.) sheets with an exceptionally smooth surface without inducing stresses common to machining, allowing the metal to be friction-bonded or brazed. Contact: 500 Fortune Blvd., Milford, MA 01757 (Chip Lehrer), phone 508/478-2023, fax 508/478-3582, website www.photofabrication.com.

* The Breakthrough Technologies Institute, Washington, DC is publishing the spring edition of its semi-annual Fuel Cells 2000 Fuel Cell Directory. It contains more than 620 listings of fuel cell manufacturers, researchers and consultants, suppliers, utilities, associations and relevant government agencies around the world. The directory costs \$50. Beginning this fall, the directory will be published annually. Contact: Bernie Geyer or Bob Rose, 202/785-9620, e-mail fuelcell-owher@analst.com; bernie@fuelcells.org.

* A Japanese researcher, Prof. Masuo Okada, of Tohoku University, in Sendai, says he has developed new vanadium-based metal hydride alloys with a storage capacity of nearly 2.5-2.6 % by weight. The new alloys have a body-centered crystal (BCC) structure and consist largely of inexpensive chromium plus 1/2 to 1/4 of Vanadium. This capacity, Okada says, is almost twice as much as with rare-earth alloys, such as MnNi₅ materials. In a recent e-mail message to H&FCL, Okada said the competition in Japan to develop materials that can store 3% hydrogen - a minimum requirement for fuel cell cars, according to him - is strong, and he expects somebody will reach that goal within a couple of years. Okada recently formed new hydride research group (H&FCL Feb. 99). Contact: Masuo Okada, e-mail okadamasa@material.tohoku.ac.jp.

* Chemical Market Associates, Inc. (CMAI), Houston, TX has announced publication of its new "World Methanol Cost Study." The study examines production and delivered costs to major markets today, with forecasts to 2003. CMAI was co-sponsor of the recent Washington methanol conference (H&FCL March 99). Prices of the study range from \$6,000 for non-clients to \$3,500 (Continued on p.8)

the Hydrogen & Fuel Cell Letter

"Alternative Energy News Since 1986"

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Five Years in the Making, \$18 Million Hydrogen Production/Fueling Station Opens in Munich

MUNICH - It's no longer the traditional question of "regular or super?" when refueling at Munich airport's new alternative energy filling station.

Rather, it's "liquid or gaseous?" at the DM 34 million (\$18.7 million), hydrogen production and refueling station inaugurated May 5 by Bavaria's minister for economics, transportation and technology, Dr. Otto Wieselhuber.

It is Germany's second hydrogen fueling station. Like the one in Hamburg that opened in mid-January (H&FCL Feb. 99), the station, conceived and built by 13 companies with the help of the state of Bavaria, is open and accessible to the public.



Munich airport's new hydrogen filling station: Shown here are a MAN articulated bus and the BMW sedan that will ferry passengers between the terminal and planes. Another 15 sedans are expected to be added next year for "Expo 2000." □

Launched about five years ago (THL Nov. 94) as a feasibility study by Deutsche Aerospace (DASA) and Munich airport planners, the gas station dispenses both fuels: gaseous hydrogen at DM 0.65 (\$0.34) per standard cubic meter, and liquid hydrogen at DM 1.10 (\$0.59 cents) per liter. Aral, Germany's leading service station chain, says that price barely covers the production cost of liquid hydrogen, and its energy content is equivalent to about

(Continued on p. 6)

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NJ PEM Fuel Cell "Venturer" Places Second in Class, First in Greenness, in Tour de Sol

LAKE GEORGE, NY - The "New Jersey Venturer," the first fuel cell vehicle ever to enter the annual American Tour de Sol U.S. Electric Vehicle Championship, finished second in the Hybrid category here in late May, beating out 9 other entries in that division the first time out (H&FCL Jan. 99).

Moreover, the car, a Solarcis/GM Geo electric modified with a small 4 kW H Power Corp hydrogen-fueled PEM fuel cell as on-board battery charger, scored highest in the division's "green" category based on emissions and fuel efficiency, according to the Tour's media office. The 11th annual tour was organized by the Northeast Sustainable Energy Association, Greenfield, MA.

Winner in the Hybrid category was a student-engineered GeoMetro from the University of Tulsa powered by a gasoline engine and an electric motor.

On overall points, the New Jersey Venturer, built by a team of volunteers from some 20 companies, government agencies and high schools just before the race, placed 13th. Overall winner on points was the Sango purpose-built electric vehicle by students from the New Hampshire Technical Institute, operating on Ovonic nickel-metal hydride batteries.

(Continued on p. 8)

Nissan Enters Fuel Cell Sweepstakes, Rolls Out Methanol/PEM Demonstrator

TOKYO - Nissan Motor Co. formally entered the race to market a fuel cell vehicle in May with the unveiling of a demonstration model powered by methanol.

Based on the R'nessa sport-utility vehicle, the hybrid model is equipped with a methanol reformer, a PEM fuel cell and lithium ion battery. A version of the standard car is being test-marketed in California.

The fuel cell was supplied by Ballard Power Systems. However, Nissan spokesman Takashi Orawa says no decision has been made on whether a Ballard fuel cell will be adopted for the commercial model.

The automaker, which presented a concept of the vehicle at the 1997 Tokyo Motor Show, plans to commercialize the model in 2003 or 2004, though only for limited usage for fleet-testing purposes by power utilities and research institutes.

(Continued on p. 8)